

Element Analysis with Fundamental Parameters using an XRF Spectrum Analysis MATLAB Algorithm

Girish Balasubramanian, Senthil Arumugam Muthukumaraswamy

Abstract— This paper proposes a MATLAB algorithm which can perform X-ray Fluorescence (XRF) spectrum analysis using a type of calibration method known as fundamental parameters (FP). This calibration method is unique because it uses the theoretical relationship between measured X-ray intensities and concentrations of the elements in a given sample. This is different from an empirical approach which uses known sample spectra and known composition and results to obtain results. The fundamental parameter approach is more complex compared to the empirical approach as there are different factors involved in the calculation of these parameters. In this paper, An XRF spectrometer (X-MET8000 by Oxford Instruments) was used for performing experimental gathering of sample spectra. Sample spectra for various pure elements and alloys were obtained and used as an input to the proposed MATLAB algorithm. The MATLAB algorithm uses the FP approach to qualitatively and quantitatively identify the elements present in the tested spectra. The proposed algorithm was able to successfully identify the elements present in the sample as well the elemental composition by means of the FP approach. This algorithm was also compared with results from another similar algorithm that was used to identify alloys by empirical means. The results obtained which include the elemental composition and the present elements is then compared with standardised known test samples of various pure elements and alloys for verification as a measure of accuracy and validity of the MATLAB algorithm which was found to be accurate to within 0.6 percentage points. The working speed of the MATLAB algorithm was also tested experimentally and was observed to be able to process 11-82 samples per second depending on the mix of the sample and type of analysis.

Index Terms— Element analysis, Fundamental parameters, Spectroscopy, Spectrum analysis, Testing algorithm, X-ray fluorescence,

I. INTRODUCTION

Testing, quality control and identification are a necessity in many applications. These can include anything from forensics, metal industries, archaeology, mining and many more. Knowing the exact elemental composition of a sample can reveal important information regarding it. In industries such as manufacturing, testing can save time and money [6]. In industries such as forensics, knowing the right

composition of trace elements in a sample can help solve a case. Spectrum analysis is a widely employed technique used to identify characteristics of a spectrum to obtain useful information. In this paper, a technique was used to analyze spectra produced by using X-Ray Fluorescence (XRF).

This paper proposes an algorithm that can perform analysis of XRF spectra to qualitatively or quantitatively identify the elements present along with their composition by means of using fundamental parameters (FP). The XRF FP algorithm was implemented using MATLAB and tested by using raw spectra of various pure elements and alloys obtained through an XRF instrument after which the efficacy of the MATLAB algorithm is successfully demonstrated.

The XRF spectra were obtained by using an XRF instrument (X-MET8000 by Oxford Instruments). The instrument uses an X-Ray tube to emit high-energy radiation onto the sample that causes a fluorescent photon with a certain energy level to be emitted from each atom hit by the radiation. This is picked up by a detector, which creates a spectrum of counts vs. energy level. Each element has a characteristic energy level representing it and this can be used to identify the presence of a certain element in the sample [7].

The MATLAB algorithm was designed using the known energy level characteristics of each element to take in XRF spectra as input. It then uses peak identification and fundamental parameter (FP) techniques [20, 21] to qualitatively and quantitatively analyze the spectrum. Once the elemental composition of the sample spectrum has been identified, it shows a summary of different elements present in the spectrum.

In order to test the algorithm, a variety of known pure elements and alloys were used as samples. The X-MET8000 was then used to obtain spectra for each of these samples, after which the spectrum data was input to the script one by one and multiple runs were performed. Lastly, the results of each test were recorded and compared with the known standards while measuring the time taken to complete the identification.

In Section II, the different techniques that are used in XRF analysis are discussed. This includes both FP and empirical methods. The FP method is also discussed more in detail and how it can be calculated along with its effect on element analysis. More details on the X-MET8000, the XRF instrument used to gather spectra and how the instrument

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was used to obtain test spectra for the MATLAB algorithm are also discussed.

In Section III, the MATLAB algorithm is discussed in detail, showcasing parts of the code highlighting the implementation of the FP calibration and also the working of the algorithm. The two different modes of analysis which include qualitative and quantitative are further discussed in this section.

In Section IV, the results obtained through MATLAB tests for both qualitative and quantitative analysis of pure metals and alloys are verified by comparing them with the expected results. The speed (samples/second) of the proposed algorithm is also tested using the MATLAB profiler. The paper is then concluded in Section VI highlighting the efficacy of the proposed MATLAB algorithm.

In Section V, the MATLAB algorithm is compared with a similar algorithm that carries out element analysis by means of empirical methods. This section highlights the differences of both algorithms and when either can be used.

II. TECHNIQUE AND THEORY

2.1 X-Ray Fluorescence

X-Ray Fluorescence or XRF for short is a technique most commonly used for the analysis of elements present in different materials. This is done by measuring secondary x-ray that gets emitted by the material or sample when excited by a primary x-ray source. An XRF instrument usually consists of an exciter (the source) and a detector. The intensity and energy level of the secondary x-rays emitted from the sample or material that is being tested is measured by the detector. Each element has a characteristic energy level K or L [7]. This information can be used to identify the elemental composition of a sample.

XRF can be used for both qualitative analysis and quantitative analysis. Analysis which only involves the element present but not the composition is known as qualitative analysis. Analysis which involves the element present as well as the composition is known as quantitative analysis. Qualitative analysis generally does not require any types of calibration but quantitative analysis requires some form of calibration.

These calibration can be either empirical or by using FP [20]. In this paper, FP is used as the calibration type for the proposed algorithm. Calibrations are required because of the matrix effects caused by having multiple elements in a sample on each other. It can be inferred from the previous statement that FP is very simple when it comes to a pure metal and grows increasingly complex with multiple elements.

2.2 Fundamental Parameter Calibration

Firstly, in order to apply FP, four conditions need to be met [16]. These include,

1. The sample must be of one layer. This means that it cannot contain any type of contamination layer, paint layer or rusting of any sort.
2. It must not contain any abnormalities in shape on the test surface. It must be flat and smooth ideally.
3. The sample must not allow any incident x-ray beams to pass through it. It must be “infinite” in thickness for the x-ray beam.
4. Pure samples of elements that require quantification need to be available.

Provided the conditions are met, (1) can be used to identify the concentration of an element i in a sample [22],

$$C_i = K_i \cdot I_i \cdot M_{is} \quad (1)$$

Where K_i : Calibration coefficient

C_i : Concentration of element in sample

I_i : Intensity of element in the sample

M_{is} : Matrix correction factor

The concentration of element in a sample given by C_i represents the elemental content of element i present within. In order to apply this method, the calibration coefficient given by K_i needs to be identified for different samples. The intensity of the element I_i is also known as the number of counts obtained at the characteristic energy level of that particular element. M_{is} is the matrix correction factor which depends on the various other elements present in the sample.

For a pure sample it can be deduced that, $C_i = 1$, $M_{is} = 1$. The the calibration coefficient, K_i can then be calculated using (2),

$$K_i = \frac{1}{I_i} \quad (2)$$

The method to calculate M_{is} is complex and requires multiple iterations for various combinations. The mathematical working and details of the method can be referred to. [22]

2.3 X-MET8000

The X-MET8000 is an XRF analyzer that was used to perform experimental gathering of raw spectra for the purpose of this study. An image of this instrument can be seen in Fig.1.



Fig. 1. X-MET8000 used for gathering of sample spectra

This instrument uses a high performance x-ray tube as the source and uses a large area silicon drift detector (SDD) to obtain the spectra from a sample and can detect energy levels upto 51.33 keV using different filters that use different voltage and current settings for various elements. This is present so that light elements such as aluminum can be detected.

The instrument was used to test various pure elements and alloys with a uniform sample time setting of 15 seconds per sample. These include precious metals such as silver, gold and platinum to everyday material elements such as copper and iron. Table 1 shows the different samples for which spectra were gathered along with their known composition.

Table 1: Elements used to test the MATLAB Algorithm

Sample	Known Composition
Pure	Al
	Ag
	Au
	Cr
	Cu
	Fe
	Mn
	Ni
	Pb
	Pt
	Sn
	Ti
	W
	Zn
	Zr
Alloy	CDA836
	CDA857

For the purpose of this paper, the results for only a few of these various pure elements and alloys are shown.

III. MATLAB ALGORITHM

3.1 Working of Algorithm

The MATLAB algorithm was designed to perform both qualitative and quantitative analysis. The quantitative analysis is performed using the FP technique which was discussed in section 2.2. The basic working of the algorithm is carried out as follows:

1. The spectrum is input to the algorithm
2. The characteristic energy levels of each elements are checked for their counts
3. The presence of elements can be confirmed with peaks identified at the characteristic energy levels.

For qualitative analysis, it stops here. However, for quantitative analysis,

4. The FP method is used to calculate the concentration of each of the present element using the calibration coefficient and the matrix correction coefficient for that particular mix.

The FP method is implemented in the MATLAB algorithm by incorporating equation (1) from section 2.2. The matrix correction factors [16] were calculated using another algorithm and was used in this algorithm to obtain the desired result.

3.2 Energy Lines

From section 2.1, it is known that each element has a unique characteristic energy level [7]. The K and L energy levels for the elements that are tested with the MATLAB algorithm are shown in Table 2 [23].

Table 2: Energy levels of the test elements

Element	K - series		L - series	
	K α (keV)	K β (keV)	L α (keV)	L β (keV)
Al	1.487	1.557	-	-
Ag	22.163	26.095	-	-
Au	-	-	9.705	11.609
Cr	5.415	5.947	-	-
Cu	8.048	8.905	-	-
Fe	6.404	7.058	-	-
Mn	5.899	6.490	-	-
Ni	8.265	7.478	-	-
Pb	-	-	10.555	12.791
Pt	-	-	9.439	11.231
Sn	25.044	28.439	-	-
Ti	4.511	4.932	-	-
W	-	-	8.396	9.819
Zn	8.639	9.572	-	-
Zr	15.770	17.662	-	-

This known data was incorporated with the MATLAB algorithm in order to locate the peaks of the respective elements. The *peakfind* function in MATLAB was used to achieve this task.

IV. RESULTS AND DISCUSSION

MATLAB results are presented for some selected samples of pure elements and alloys. These include silver, iron, copper and lead for pure elements and CDA836 and CDA857 for alloys.

4.1 Qualitative Results

The first step to quantizing the results requires verification on whether the algorithm is able to successfully identify the presence of a certain element in a sample. Selections of samples were tested using the qualitative test and the spectra of these samples were input to the MATLAB algorithm. Fig.2 shows the spectrum of a known pure silver sample that was applied to the MATLAB algorithm.

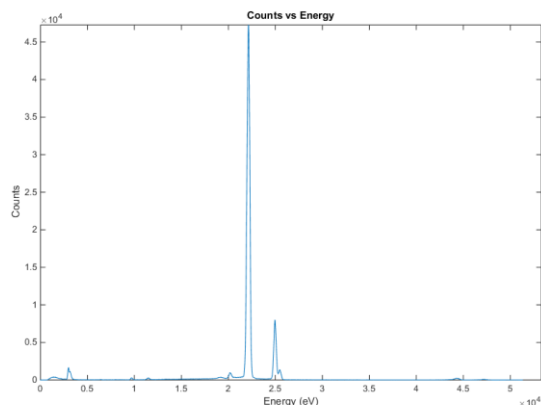


Fig. 2. Silver sample spectrum obtained through X-MET8000

The algorithm first uses the peakfind function to identify whether there are any characteristic peaks present within the spectra. Fig.3 demonstrates the result obtained when the algorithm was used to perform this function.

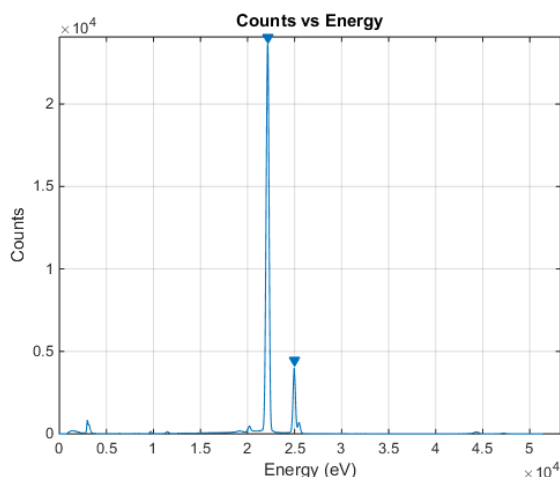


Fig. 3. Silver sample spectrum passed through the proposed algorithm with peaks identified.

It was observed that the peaks were correctly identified at 22.163 keV and 26.1 keV as per the expected theoretical energy level for silver that was shown in Table 2. Using this information, the algorithm can also identify that there are no other characteristic peaks, and hence no other element present within the spectrum. Following this development, the algorithm provides the user with a summary of what was observed over the course of the test. Fig.4 showcases this.

```
-----Starting XRF Test-----
-----Testing Spectra Qualitative-----
Silver - Yes
Elapsed time is 0.012229 seconds.
```

Fig. 4 Summary of qualitative results obtained through the MATLAB algorithm for the silver sample

Similarly, this test was performed for other pure samples including iron, lead and copper. Fig. 5, Fig. 6 and Fig. 7 shows the spectra obtained when these sample spectra were passed through the MATLAB algorithm.

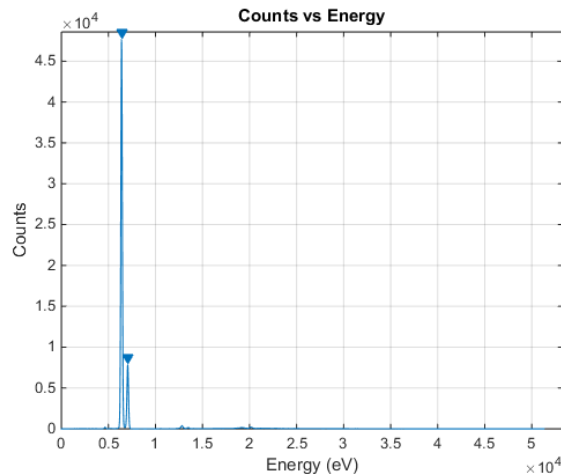


Fig. 5. Iron sample spectrum passed through the proposed algorithm with peaks identified

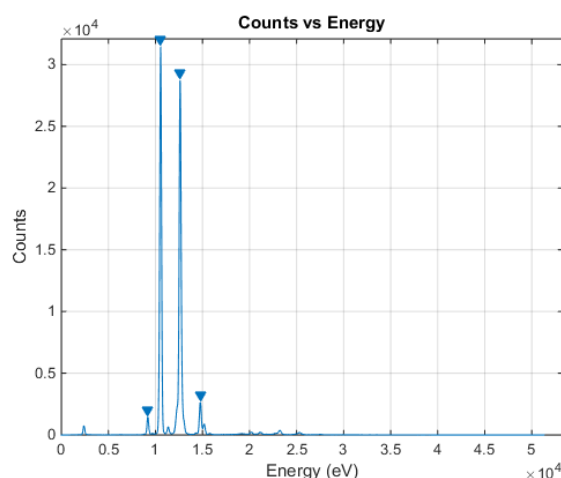


Fig. 6. Lead sample spectrum passed through the proposed algorithm with peaks identified

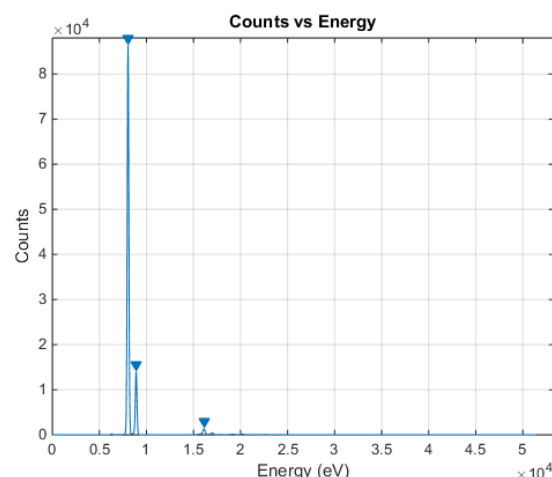


Fig. 7. Copper sample spectrum passed through the proposed algorithm with peaks identified

For some of these spectra, namely copper and lead that is shown, it was observed that the peakfind function also identified some smaller peaks that were not related to the characteristic energy level of any elements. These were disregarded when identifying the presence of elements within these spectra. The test was also performed for mix samples (alloys). The spectra for CDA836 and CDA857

obtained by using the MATLAB algorithm are shown in Fig. 8 and Fig. 9 respectively.

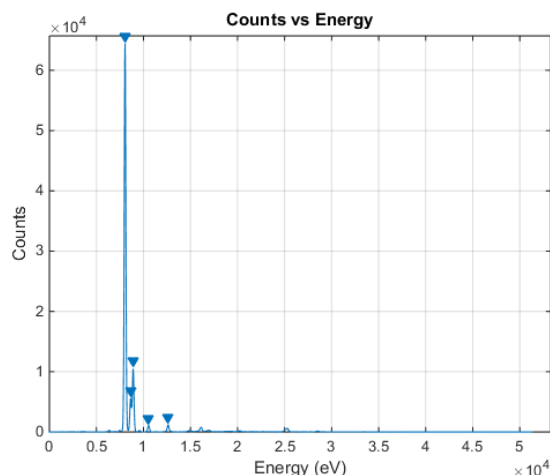


Fig. 8. CDA836 sample spectrum passed through the proposed algorithm with peaks identified

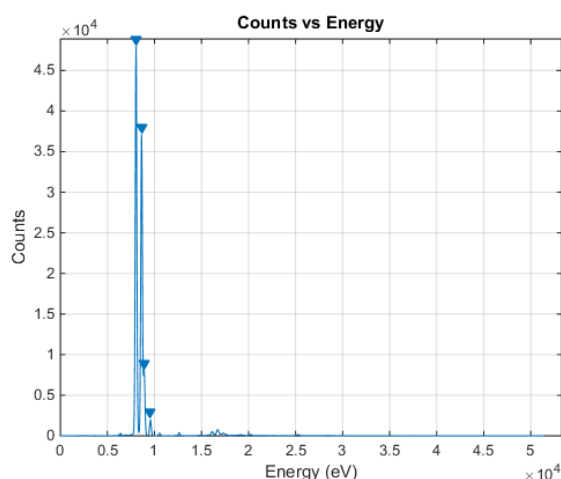


Fig. 9. CDA857 sample spectrum passed through the proposed algorithm with peaks identified

The qualitative results obtained using the MATLAB algorithm for all the samples that were tested are tabulated in Table 3.

Table 3: Results obtained through qualitative testing using the proposed MATLAB algorithm

Sample	Expected Result	Result Obtained
Pure Silver	Silver	Silver
Pure Iron	Iron	Iron
Pure Lead	Lead	Lead
Pure Copper	Copper	Copper
CDA836	Copper, Lead, Tin, Zinc	Copper, Lead, Tin, Zinc
CDA857	Copper, Lead, Zinc	Copper, Lead, Zinc

4.2 Quantitative Results

Once the results obtained from qualitative testing were observed to be satisfactory, the FP method is used to obtain the matrix correction factors each mix and hence the concentration of each element in the sample. For pure elements, this is fairly straightforward as the matrix correction factor is simply one. The result obtained for pure

iron for quantitative analysis was obtained. This can be seen in Fig. 10.

```

-----Starting XRF Test-----
-----Testing Spectra-----
Aluminium - No/Trace
Silver- No/Trace
Gold - No/Trace
Chromium - No/Trace
Copper - No/Trace
Iron - 99.9047%
Manganese - No/Trace
Nickel - No/Trace
Lead - No/Trace
Platinum - No/Trace
Tin - No/Trace
Titanium - No/Trace
Tungsten - No/Trace
Zinc - No/Trace
Zirconium - No/Trace
Traces of other elements = 0.095288%
-----End of Test-----
----- Summary of Results -----
Iron - 99.9047%
Other = 0.095288%
Elapsed time is 0.021974 seconds.

```

Fig. 10. Quantitative results obtained when a pure iron sample was input to the proposed MATLAB algorithm

Similarly, results for other samples were also obtained. Fig. 11 shows the quantitative results obtained for an alloy sample of CDA836.

```

-----Starting XRF Test-----
-----Testing Spectra-----
Aluminium - No/Trace
Silver- No/Trace
Gold - No/Trace
Chromium - No/Trace
Copper - 84.6009%
Iron - No/Trace
Manganese - No/Trace
Nickel - No/Trace
Lead - 4.9937%
Platinum - No/Trace
Tin - 4.99%
Titanium - No/Trace
Tungsten - No/Trace
Zinc - 5.0013%
Zirconium - No/Trace
Traces of other elements = 10.4502%
-----End of Test-----
----- Summary of Results -----
Copper - 84.6009%
Lead - 4.9937%
Tin - 4.99%
Zinc - 5.0013%
Other = 0.4141%
Elapsed time is 0.101641 seconds.

```

Fig. 10. Quantitative results obtained when an alloy sample of CDA 836 was input to the proposed MATLAB algorithm

These results for all the tested samples were then tabulated and displayed in Table 4.

Table 4: Results obtained for test samples for quantitative analysis using the MATLAB algorithm

Sample	Expected Result	Result Obtained
Pure Silver	99.9% Silver	Silver
Pure Iron	99.9% Iron	Iron
Pure Lead	99.9% Lead	Lead
Pure Copper	99.9% Copper	Copper
CDA836	85% Cu, 5% Pb, 5% Sn, 5% Zn	84.6% Cu, 4.99% Pb, 4.99% Sn, 5% Zn
CDA857	60.9% Cu, 1.58% Pb, 36.1% Zn	60.3% Cu, 2.04% Pb, 35.8% Zn

From these results it was observed that the proposed algorithm was successfully able to identify the elemental composition of the samples tested. The maximum error was found for CDA857 with only a minimal deviation of 0.6% points from the expected result.

3.1 Algorithm Speed

For all the tests that were run, the time taken for the algorithm to execute was measured using the MATLAB profiler. The results obtained for the tested samples are tabulated in Table 5.

Table 4: Average time taken to test each sample using the MATLAB algorithm

Sample	Time taken (Qualitative) (s/sample)	Time taken (Quantitative) (s/sample)
Pure Silver	0.0134	0.022
Pure Iron	0.01322	0.022
Pure Lead	0.0131	0.022
Pure Copper	0.01241	0.022
CDA836	0.0242	0.102
CDA857	0.0231	0.078

It was calculated that the average time taken for pure elements for qualitative analysis was roughly 0.0125 seconds/sample with various elements not limited to the ones tested in this paper, while being roughly 0.0237 seconds/sample for alloys. For quantitative analysis, it was calculated to be 0.022 seconds/sample for pure elements and roughly 0.09 seconds/sample for alloys.

V. COMPARISON WITH EMPIRICAL ALGORITHM

A similar algorithm was devised earlier [23] that was used to identify elements for metals by empirical means. This meant that the algorithm could only be used to check whether the sample matched a previously known sample present in the database to a certain degree. This introduces great limitations when it comes to element identification. A significant limitation is that the empirical algorithm would not be able to identify the composition or presence of elements that is not already present in the alloy database. A great deal of work needs to be done to populate a large database and it would be difficult for many industries or even small-scale users to obtain such a database.

The advantage that this proposed algorithm provides is the ability to perform analysis on samples without the need for a database but using theoretical relationships between the elements. This is done using the FP approach discussed earlier. There is still a limitation to the FP approach as pure samples still need to be available to carry out the FP method. However, the elimination of requiring a database or a known benchmark sample to obtain an analysis is eliminated.

This comes at a cost of speed for pass/fail as the empirical algorithm provides a greater speed and samples that can be handled in a given time period for pass/fail mode. Some industries that perform bulk sample testing of similar subjects may benefit from the empirical algorithm more as it is almost three times faster than the FP algorithm proposed in this paper. However, the alloy identification mode would take a much longer time as it has to compare with an entire database of alloys. This is another drawback to the empirical algorithm where the identification of the sample would be slower than the FP algorithm if the database used is very large.

There are applications where accuracy and benchmark samples may not be readily available. These include archaeology, mining, etc. In these applications the FP method can prove to provide accurate and reliable results especially since time is not as much of a factor as compared to large-scale metal industries.

Another significant difference between both algorithms is the inability to perform quantitative analysis in the previously proposed algorithm [23]. The algorithm proposed in this paper provides element composition results accurate to within 0.6 percentage points. However, the empirical algorithm was only able to identify whether there is a match with the alloys present within a pre-existing database.

VI. CONCLUSION

The MATLAB algorithm used was able to execute the FP XRF analyzer accurately while producing the expected results reliably. This was observed from the results obtained for both qualitative and quantitative analysis. The FP method was deemed to be a successful approach to obtaining the element composition present in a sample. The proposed MATLAB algorithm can be further specialized into any required application.

The qualitative part of the algorithm was able to identify spectra of silver, iron, copper for pure elements as well as CDA836 and CDA857 when it came to alloys. The results show that the algorithm gave a "yes" for all the elements that were expected to be present in the tested samples. Moreover, the results show that the time taken to carry out each test averaged to around 0.0125 seconds/sample for pure elements and 0.0237 seconds/sample for alloys. The quantitative part of the algorithm was then able to correctly extend the qualitative part by using the present element information in the FP calculations to correctly identify the % of each of the present elements in the sample. The results show that the maximum deviation was 0.6 percentage points for copper in CDA857. This is a minimal difference and does not change the fact that the algorithm was able to correctly identify the elemental composition of all of the test samples. Moreover, performing the quantitative test averaged at around 0.022

seconds/sample for pure elements and 0.09 seconds/sample for alloys.

Comparing with the MATLAB algorithm that was designed using an empirical approach, the algorithm proposed in this paper is shown to provide composition data but being slower by 0.34 seconds on average when comparing alloy identification directly to each other. However, this result is actually much better when keeping in mind that the database size has to be considered. This is a big improvement in many applications and is only a downgrade to applications where bulk testing is required of similar metals with a small database.

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